Appendix S

Porosity Tests—Part 2

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Porosity Tests—Part 2

The porosity of a material is the ratio of void volume over the total volume of a sample. Diffusive movement of contaminants is controlled by availability of pore space in the waste form through which the contaminant can diffuse. Both aqueous- and vapor-phase diffusion are controlled by the presence of water. Therefore, porosity and moisture characteristic curves are important parameters in mechanistic modeling of contaminant release from treated waste.

Porosity is used in mechanistic modeling of release from treated waste and is a fundamental modeling input that controls both the interstitial velocity at which a given Darcian water flux moves and the diffusive fluxes in both the aqueous and gaseous phases. The porosity of a porous medium is the ratio of volume of void spaces to total sample volume.

S-1. TEST OBJECTIVES AND RATIONALE

The purpose of these tests was to compare a specifically formulated proprietary grout with commercially available grouts in terms of their ability to maintain compressive strength, decrease hydraulic conductivity and porosity, and reduce contaminant mobility when mixed with surrogates of anticipated waste streams. Porosity measurements were made on neat grout samples, 70-30 wt% grout-soil samples, and 50-50 wt% grout-soil samples. Three grout mixtures were tested: GMENT-12, Portland, and a mixture of 50 wt% Portland and 50 wt% blast furnace slag (see Table S-1).

Table S-1. Summary of porosity testing for waste from nontransuranic pits and trenches and soil vault rows.

Method	Waste Matrix	Grouts	Waste in Grout (wt%)	Replicates	Total Samples
Porosity test for aggregate concrete (ASTM D2434-68)	Soil from the Idaho National Laboratory Site	GMENT-12	0	3	27
		Portland	30		
		Portland/slag	50		

S-2. EXPERIMENTAL DESIGN AND PROCEDURES

To determine the volume of void spaces in these porous media, voids must be filled completely with water so that the fluid can be measured. By flushing the samples with carbon dioxide gas and then saturating the samples with deaerated water under a vacuum, the pores can be filled with water in the absence of entrapped air, allowing calculation of sample porosity (Dane and Topp 2002). The ASTM standard for saturating samples to determine hydraulic conductivity (ASTM D2434-68) and methods described in Dane and Topp were referenced for direct measurements of porosity. In consolidated porous media, measured porosity is a measurement of the void space of interconnected pores. Dead-end pores are those with no connection either to other pores or to the outside of the sample. These dead-end pores will remain void of water even during saturation. Dead-end pores are not included in this measurement since water cannot be imbibed into the pores during sample saturation.

Estimated or calculated porosity from sample dry bulk density measurements includes the void space of all pores in the sample (Jury, Gardner, and Gardner 1991). Differences between calculated and measured porosity may be because of uncertainties in material or particle density values or because there are dead-end pores in the sample.

Samples were prepared in cylinders that are 5.5 cm (2.17 in.) tall by 3.25 cm (1.28 in.) in diameter as shown in Figure S-1. Volumes of the samples were determined using a water displacement method. Samples were allowed to cure 30 days at 98–99% humidity in an environmental chamber. The water used in porosity measurements was an Idaho National Laboratory (INL) Site groundwater surrogate (see Appendix B).

Figure S-1. Examples of porosity samples.

S-3. EQUIPMENT AND MATERIALS

Materials used to saturate the samples include a vacuum desiccator, a 600-mm (23.6-in.) mercury vacuum pump, a carbon dioxide tank, INL Site synthetic groundwater, and a water deaerator (see Figure S-2). The INL Site synthetic groundwater was deaerated by autoclaving the water in a 7.6-L (2-gal) carboy at 121ºC (249.8ºF) and 21 psi for 25 minutes. Water in the carboy was allowed to cool to room temperature while sealed to prevent aeration. The samples were placed in the vacuum desiccator. The vacuum desiccator was evacuated for 5 minutes, flushed with carbon dioxide for 5 minutes, and then evacuated again to remove all gas from the chamber. The process of evacuating and carbon dioxide flushing was repeated three times, and then the samples were saturated under 500 mm (19.7 in.) of mercury from the bottom up to allow entrapped soil gas to escape at sample surface during water imbibition.

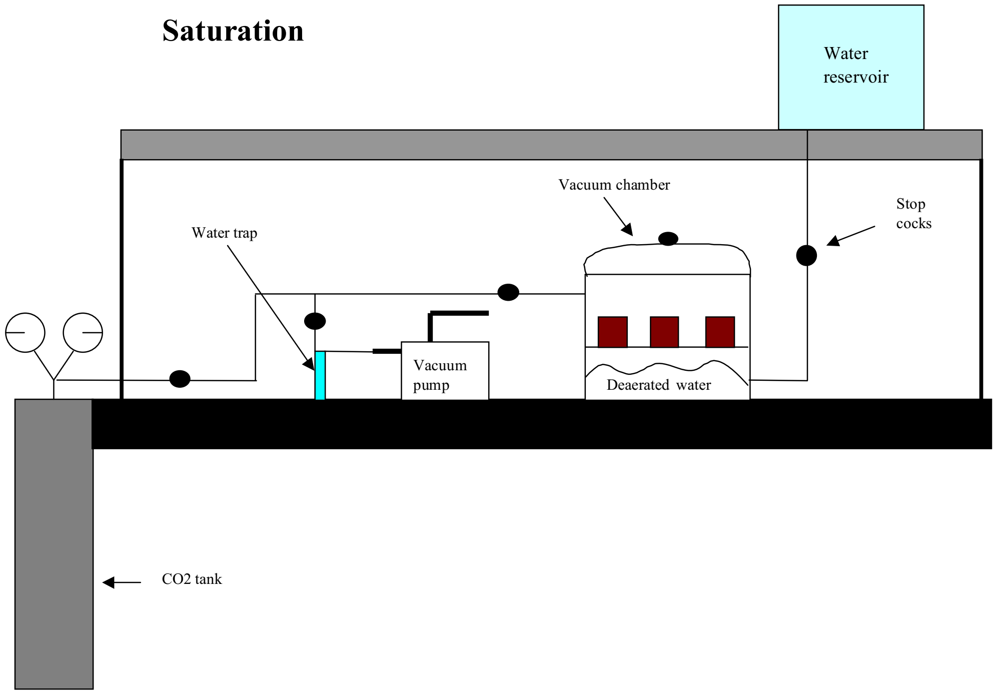

Figure S-2. Saturation chamber, vacuum pump, and carbon dioxide tank.

Once the water level reached midway up the sample length, the chamber was sealed, and the samples were left in the chamber for 24 hours under a vacuum to allow the sample to reach complete saturation. After 24 hours, the samples were removed from the chamber, and excess water around the edges of the samples was wiped off, taking care not to wick up any water from the sample pores. The sample was weighed to determine the mass of the imbibed water. The samples were then placed in a drying oven for 24 hours to remove the water. The samples were weighed again. The volume of the samples was determined using water displacement. The volume of water was calculated from the sample water mass, assuming a density of 1 g/cm^3 for the water (wet weight – dry weight). Porosity was determined by dividing volume of water in the sample (wet sample weight – dry sample weight) by total sample volume.

S-4. DATA MANAGEMENT, ANALYSIS, AND INTERPRETATION

Data were recorded in a laboratory notebook, which includes procedures, raw data, and some calculations. The majority of the calculations were performed and recorded in an Excel spreadsheet, which can be provided on request.

Figure S-3 shows results of porosity tests. Error bars represent the 95% confidence interval. The data are listed in Table S-2. Measured values of porosity for surface soil from the Idaho National Laboratory Site range from approximately 0.4 to 0.5 and are represented by the line shown in Figure S-3 at 0.45.

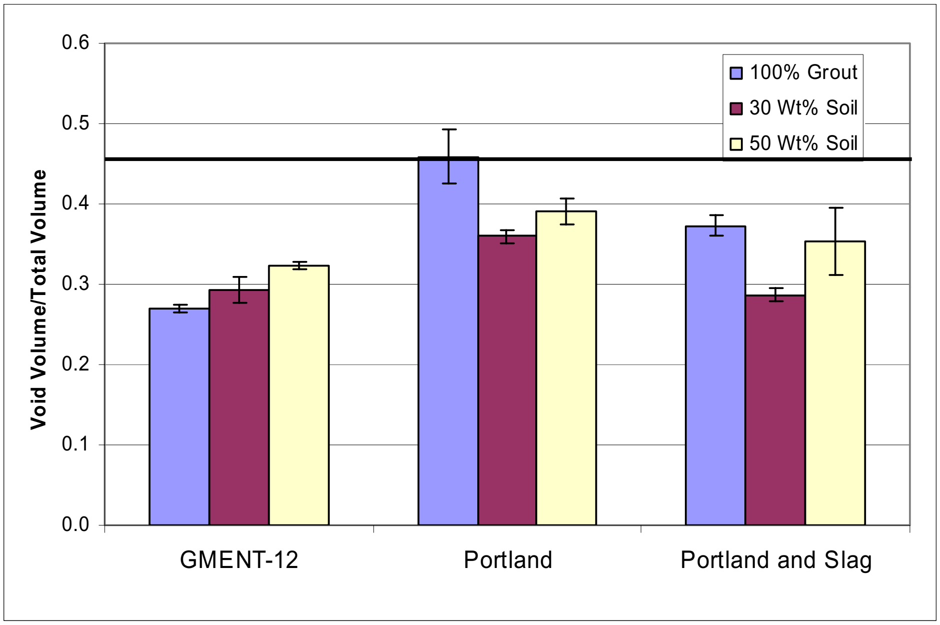

Figure S-3. Porosity of grouted waste forms.

Table S-2. Sample porosity for neat grout and soil from the Idaho National Laboratory Site.

	Porosity of Grouted Waste Forms (mL/mL)		
Grout Type	Neat Grout	Soil from the Idaho National Laboratory Site (30 wt%)	Soil from the Idaho National Laboratory Site (30 wt%)
GMENT-12			
Specimen A	0.268	0.285	0.325
Specimen B	0.270	0.295	0.321
Specimen C	0.271	0.298	0.323
Mean	0.270	0.293	0.323
Standard deviation	1.47E-03	6.66E-03	1.71E-03
95% confidence interval	3.66E-03	1.65E-02	4.25E-03
Portland			
Specimen A	0.447	0.362	0.396
Specimen B	0.454	0.362	0.391
Specimen C	0.474	0.356	0.383
Mean	0.458	0.360	0.390
Standard deviation	1.36E-02	3.27E-03	6.60E-03
95% confidence interval	3.37E-02	8.12E-03	1.64E-02
Portland/slag			
Specimen A	0.367	0.290	0.350
Specimen B	0.378	0.286	0.339
Specimen C	0.374	0.284	0.372
Mean	0.373	0.287	0.354
Standard deviation	5.18E-03	3.22E-03	1.70E-02
95% confidence interval	1.29E-02	8.00E-03	4.22E-02

S-5. RESULTS AND CONCLUSIONS

In all cases, the grouts mixed with soil produced porosity values less than the estimated porosity of the soil alone, as shown by the dark line in Figure S-3.

When tested as neat grout, the porosity of GMENT-12 was significantly lower than the other two grouts. Porosity of GMENT-12 samples increased as the percent soil added increased, just as with hydraulic conductivity (see Appendix T).

When Portland grout was tested, porosity of the neat grout was significantly higher than when soil was added at both the 30 and 50 wt% loading. Adding soil to the grout decreased porosity of the sample. Again, this same condition was demonstrated when testing the hydraulic conductivity of the samples. When the mixture of Portland cement and slag was tested, the 70 wt% grout loading had a significantly lower porosity than either the neat Portland cement with slag or the 50 wt% loading.

Based on porosity results, the commercially available grouts (Portland cement or Portland cement with blast furnace slag) performed at least as well as the proprietary GMENT-12 and in some cases better.

S-6. REFERENCES

ASTM D2434-68, 2000, "Standard Test Method for Permeability of Granular Soils (Constant Head)," ASTM International.

Dane, J. H. and G. C. Topp, eds., 2002, *Methods of Soil Analysis Part 4 Physical Methods*, Madison: Soil Science Society of America, Inc.

Jury, W. A., W. R. Gardner, and W. H. Gardner, 1991, *Soil Physics*, New York: John Wiley and Sons, Inc., 328 pp.

Appendix T

Hydraulic Conductivity Tests—Part 2

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Hydraulic Conductivity Tests—Part 2

Hydraulic conductivity describes the ability of a porous medium to transmit fluids. The magnitude of hydraulic conductivity depends primarily on size distribution, roughness, tortuosity, shape, and degree of interconnection of the fluid-conducting pores. Because hydraulic conductivity is highly sensitive to texture and structure of the porous medium, mixing low-permeability grout materials into soil and soil-waste mixtures should significantly reduce the hydraulic conductivity of the waste forms, therefore reducing mobility of radioactive and hazardous contaminants in waste buried at the Subsurface Disposal Area.

These hydraulic conductivity tests help evaluate the potential application of in situ grouting to remediate low-level waste pits and trenches and soil vault rows. Comparison of test results demonstrates relative efficiency in reducing hydraulic conductivity and potential for contaminant transport. Data also will be used to evaluate long-term physical stability of grouted waste forms. Data will support modeling to estimate the release rate of contaminants from treated waste and compare with the predicted release rate of untreated waste.

T-1. TEST OBJECTIVES, RATIONALE, AND DEVIATIONS FROM TEST PLAN

Two general laboratory methods are used for determining saturated hydraulic conductivity of porous materials: constant head (for porous media with high permeability) and falling head (for porous media with low permeability). Column permeameters are used in both procedures. Because samples for this study are expected to have low permeability, the falling-head method using a flexible wall permeameter was selected originally to measure hydraulic conductivity of neat grouts and of mixtures of waste and grout (ASTM 5084-00).

The flexible wall permeameter accurately measures flow through low-permeability porous media because flow along the edges of the sample holder is prevented by pressure applied along the sample holder flexible walls. However, the disadvantage of this method is that it is time intensive. To reduce the measurement time, ASTM 5084-00 was modified based on Dane and Topp (2002). Instead of using flexible wall permeameters, the modified method uses Delrin plastic sleeves. Sample molds were prepared and then sealed into the plastic sleeves using epoxy (J. B. Weld from Sulphur Springs, Texas) to prevent preferential flow along the sample edges.

T-2. MATERIALS AND METHODS

Three grouts were investigated for immobilization of subsurface contaminants at the Idaho National Laboratory (INL) Site. The grouts include GMENT-12, Portland, and a combination of Portland and ASTM International-certified blast furnace slag (mixed at a ratio of 50-to-50). Uncontaminated soil from the INL Site was used as the surrogate to simulate contaminated soil in the nontransuranic pits and trenches and soil vault rows. Loomis et al. (2003) previously tested hydraulic conductivity of the surrogate mixtures for organic sludge, nitrate sludge, and soil from the INL Site and three of the neat grouts (GMENT-12, U.S. Grout, and TECT HG). These data along with results from hydraulic conductivity testing with WAXFIX with surrogates for organic sludge, nitrate salt sludge, soil from the INL Site, and thermally treated organic sludge are presented in Appendix D. This appendix describes the bench tests performed using more commercially available grout types (Portland cement and Portland cement with blast furnace slag) with soil from the INL Site.

T-3. SAMPLE PREPARATION

Three grout types were tested at three waste loadings. The grouts were GMENT 12, Portland, and a combination of Portland and ASTM International-certified blast furnace slag. Grout and waste mixtures were prepared according to predetermined maximum loading percentages to maintain cohesive contiguous columns and an adequate compressive strength of 250 psi (see Table T-1). The samples were cured for at least 30 days at 98–99% humidity in a constant humidity environmental chamber. When cured, they were removed from the molds, the edges were roughed, and the samples were sealed into plastic sample holders using epoxy (J. B. Weld, from Sulphur Springs, Texas).

Table T-1. Summary of hydraulic conductivity testing for cold surrogates using in situ grouting of waste from transuranic pits and trenches.

Test Method	Measurement or Analytical Method	Waste Matrix	Grout	Waste in Grout (wt%)	Replicates	Total Samples
ASTM 5084-00	Falling-head method	Soil from the INL Site	GMENT-12	0	3	27
			Portland	30		
			Portland/slag	70		

T-4. TEST DESIGN AND PROCEDURES

In a falling-head permeameter, water is introduced to a saturated sample column by gravity drainage from a burette or standpipe, while the head on the downstream end remains constant (see Figure T-1). To prevent preferential flow along the edge of sample holders using the falling-head method, the samples were epoxied into Delrin plastic sample rings measuring 25 mm (1 in.) in diameter by 45 mm (1.7 in.) high (see Figure T-2).

To test for preferential flow along the edges of the rings, samples were pretested using red dye before starting the conductivity tests. The samples were then saturated under a vacuum (see Appendix C). A plastic cap with a water-saturated 1/2-bar ceramic-disk insert was placed on top of the sample, sealing with an O-ring to the sample ring. The ceramic disk disbursed the water uniformly across the top of the sample during infiltration. Permeability of the disk was greater than the sample material and, thus, did not influence measurement results.

A 10-mm (0.4-in.)-diameter burette was attached to the top of the sample cap. All air was removed from the porous-ceramic disk, the cap, and the tubing connecting the cap to the burette. The sample was placed in a beaker of water, and the burette was filled with water to the 0-mL mark. The initial distance from the water level in the burette and the water in the beaker was determined for each sample (ΔH). Permeameters were placed in a humidity chamber to minimize evaporation during testing. A blank permeameter (no waste form) tested daily evaporation rates inside the humidity chamber.

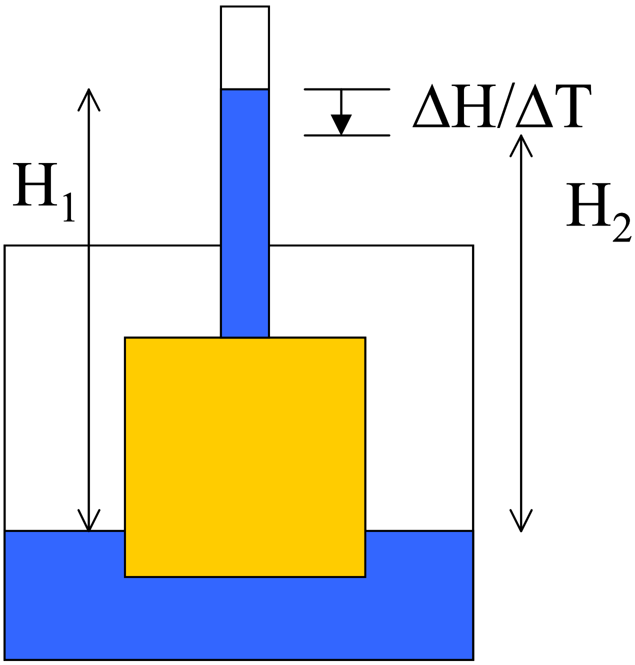

Figure T-1. Falling-head method for measuring hydraulic conductivity of low-permeability porous media.

Figure T-2. Delrin plastic molds, 25 mm (1 in.) in diameter by 45 mm (1.7 in.) high, used as sample holders for the waste forms.

Figure T-3 shows the experimental setup and instrumentation used for falling-head tests of all the grouts and grout-waste mixtures in this study. The water level in the burette was monitored over a 1-week period, and changes in hydraulic head and changes in time were recorded periodically during the test. Data representing steady-state conditions (i.e., constant flux and hydraulic gradient) were used to calculate hydraulic conductivity.

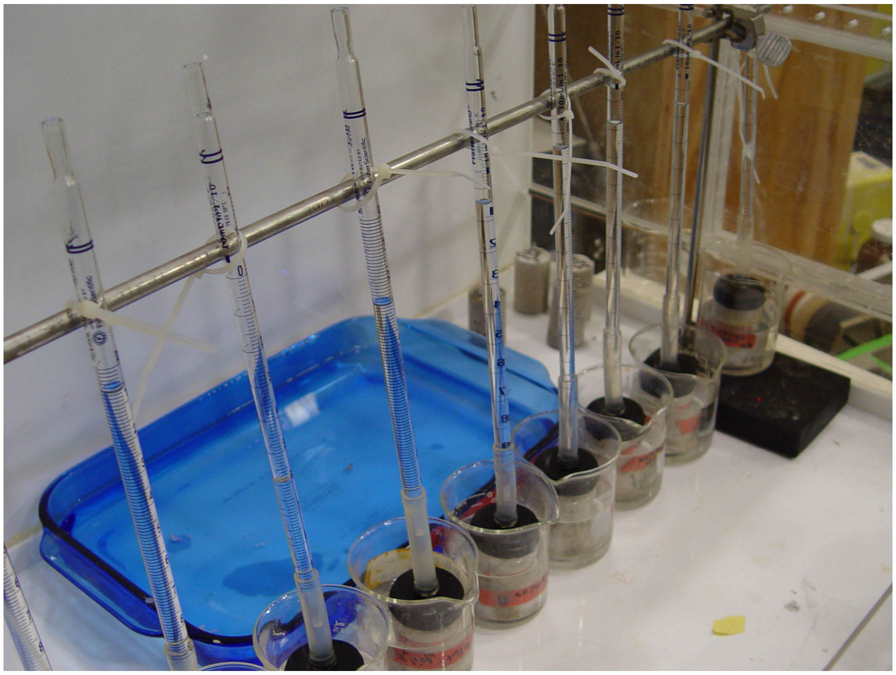

Figure T-3. Photograph of hydraulic conductivity measurement in 98 to 99% humidity-controlled environmental chamber.

T-5. DATA MANAGEMENT, ANALYSIS, AND INTERPRETATION

Data were recorded in a laboratory notebook, which includes procedures, raw data, and some calculations. The majority of the calculations were performed and recorded in an Excel spreadsheet, which can be provided on request. The hydraulic conductivity of a porous material is qualitatively defined as the ability of a saturated porous medium to transmit fluids. The physical relationship often used to describe fluid flow through porous materials is Darcy's Law (Dane and Topp 2002). The saturated hydraulic conductivity of a porous medium can be calculated from direct measurements of flux, hydraulic head, and column dimensions.

Modification of Darcy's Law is often used to calculate hydraulic conductivity using the falling-head method (Stephens 1995) as shown in Equation (T-1):

$$K = \frac{aL}{At} \ln\left(\frac{H_1}{H_2}\right) \tag{T-1}$$

where:

A	=	sample cross-sectional area [L2]
L	=	sample length [L]
a	=	burette's cross-sectional area [L2]
t	=	time between measurements
H_1 and H_2	=	water levels in the burette at t1 and t2, respectively.

Data were quality checked before being entered into an Excel spreadsheet, then were checked again after entering.

T-6. RESULTS AND CONCLUSIONS

The GMENT-12 grout mixed with soil displayed the classic example for the expected outcome for hydraulic conductivity with increasing interference (i.e., soil) added (i.e., as the concentration of interference increased, the hydraulic conductivity increased) (see Figure T-4). However, hydraulic conductivity for Portland samples decreased when soil was added at a ratio of 70 wt% grout to 30 wt% soil, but increased to the same hydraulic conductivity as the neat grout when 50 wt% soil was added. When blast furnace slag was added to the Portland cement, the hydraulic conductivity decreased with increasing soil loading up to 50 wt% soil. Interestingly, GMENT-12 had the lowest hydraulic conductivity for the three grouts when tested as neat grout and the highest hydraulic conductivity when mixed with 50 wt% soil. Conversely, the Portland cement and blast furnace slag had the highest hydraulic conductivity when tested as neat, but the lowest hydraulic conductivity when mixed with 50 wt% soil.

The porosity tests behaved in a similar manner (see Appendix S). Porosity of GMENT-12 increased with increasing soil added, and porosity of Portland cement and Portland cement with blast furnace slag decreased when soil was added at 30 wt%, then increased only slightly when 50 wt% soil was added.

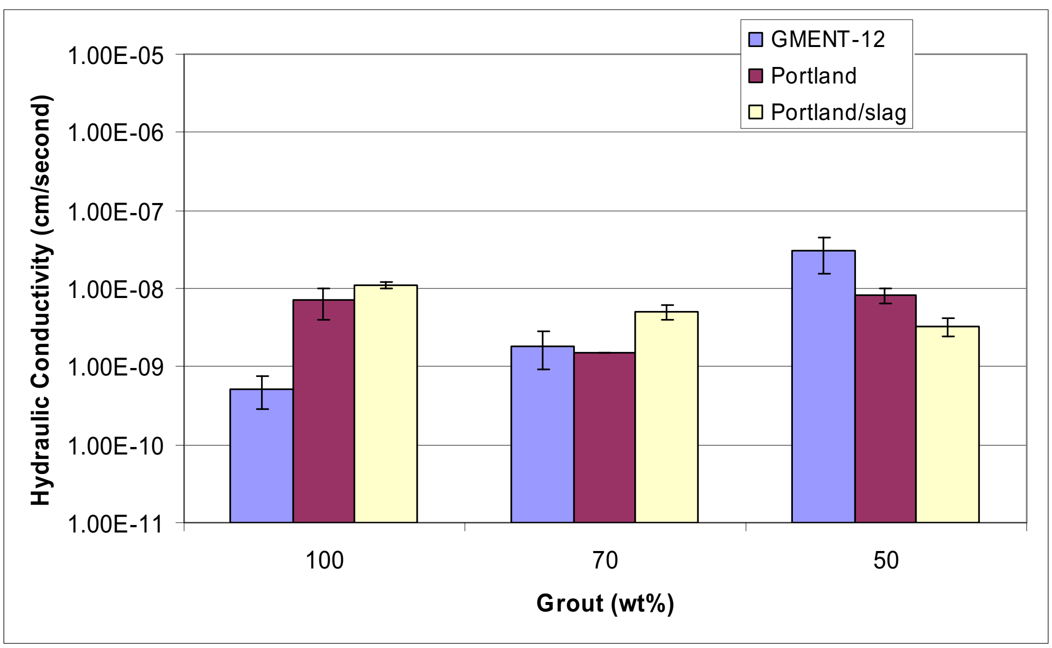

Figure T-4. Hydraulic conductivity measurements for three grout types.

Based on these results, all of the grouts behaved satisfactorily with respect to hydraulic conductivity (see Table T-2). Portland mixed with blast furnace slag provides the greatest reduction in hydraulic conductivity at 50 wt%, while GMENT-12 and Portland cement performed equally well with 30 wt% soil added.

Table T-2. Hydraulic conductivity values for grouted samples.

	Grout Product		
Grout Type	GMENT-12	Portland	Portland and Blast Furnace Slag
Neat Grout			
Specimen A	2.56E-09	5.47E-09	9.05E-09
Specimen B	1.71E-09	3.09E-09	9.79E-09
Specimen C	2.65E-09	2.31E-08	1.43E-08
Specimen D	N/A	2.43E-09	N/A
Specimen E	N/A	8.05E-10	N/A
Mean	2.31E-09	6.99E-09	1.10E-08
Standard deviation	5.18E-10	9.18E-09	2.84E-09
95% confidence interval	2.35E-10	3.00E-09	1.18E-09
30 wt% soil from the INL Site			
Specimen A	3.10E-09	1.49E-09	6.30E-09
Specimen B	7.61E-10	1.51E-09	1.87E-09
Specimen C	4.42E-09	1.49E-09	6.89E-09
Mean	2.76E-09	1.50E-09	5.02E-09
Standard deviation	1.85E-09	5.84E-12	2.74E-09
95% confidence interval	9.17E-10	2.48E-12	1.09E-09
50 wt% soil from the INL Site			
Specimen A	6.25E-08	1.31E-08	9.17E-10
Specimen B	2.24E-07	1.57E-07	4.52E-09
Specimen C	4.17E-08	3.05E-08	4.29E-09
Mean	1.09E-07	6.69E-08	3.24E-09
Standard deviation	9.98E-08	7.86E-08	2.02E-09
95% confidence interval	4.94E-08	3.51E-08	8.56E-10

T-7. REFERENCES

ASTM 5084-00, 2001, “Standard Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter,” ASTM International.

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Loomis, Guy G., James J. Jessmore, Jerry R. Weidner, Christopher M. Miller, and Allen L. Sehn, 2003, *Final Results Report, In Situ Grouting Technology for Application in Buried Transuranic Waste Sites*, INEEL/EXT-02-00233, Rev. 1, Idaho National Engineering and Environmental Laboratory.

Stephens, D. B., 1995, *Vadose Zone Hydrology*, Boca Raton: Lewis Publishers, pp. 347.